*भारतीय मानक*

***Indian Standard***

**IS 3025 (Part 59) : 2023**

# जल एवं अपशिष्ट जल के नमूने लेने और परीक्षण (भौतिक एवं रसायन) की पद्धतियाँ

## भाग 59 मैंगनीज

*( दूसरा पुनरीक्षण )*

# Methods of Sampling and Test (Physical and Chemical) for Water and Wastewater

## Part 59 Manganese

*( Second Revision )*

ICS 13.060.50

भारतीय मानक ब्यूरो
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Water Quality Sectional Committee, CHD 36

## FOREWORD

This Indian Standard (Part 59) (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Water Quality Sectional Committee had been approved by the Chemical Division Council.

Manganese is a naturally occurring element that is found in rock, soil, water and food. It usually occurs together with iron. Surface water (rivers and reservoirs) do not usually contain high concentrations of manganese because the oxygen rich water enables the mineral to settle out as sediments often associated with micro-organisms and as complexes with, for example, humic acid. In deep wells and springs where the oxygen content and *p*H tend to be low, all manganese will be present in dissolved forms and water containing manganese appears colourless.

Manganese can affect the flavour and colour of water and can also react with tannins present in beverages to form a black sludge affecting both the taste and appearance.

The Committee responsible for the formulation of IS 3025 : 1964 ‘Methods of sampling and test (physical and chemical) for water used in industry, had decided to revise the standard and publish it as separate parts. This standard is one of the different parts under the IS 3025 series of standards and superseded **35** of IS 3025. The first revision of this standard was published in 2006.

In this revision the following changes have been incorporated:

a) Periodate calorimetric method has been deleted;

b) Formaldoxime photometric method has been deleted;

c) Direct air-acetylene flame atomic absorption spectrometry (AAS) method has been added;

d) Persulphate method has been incorporated; and

e) Inductively coupled plasma spectroscopy methods have been added.

In the preparation of this standard, considerable assistance has been derived from the Method No. 3111 B, and 3500 Mn –A, B of — Standard methods for the examination of water and wastewater, published by the American Public Health Association, Washington, USA, 23rd Edition, 2017.

The composition of the Committee responsible for formulation of this standard is given in Annex A.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 2022 ‘Rules for rounding off numerical values (*second revision*).’

*Indian Standard*

# METHODS OF SAMPLING AND TEST (PHYSICAL AND CHEMICAL) FOR WATER AND WASTEWATER

## PART 59 MANGANESE

*( Second Revision )*

## 1 SCOPE

This standard (Part 59) prescribes three methods as follows for determination of manganese in water and wastewater:

a) Direct air-acetylene flame atomic absorption spectrometry (AAS) method;

b) Persulphate method; and

c) Inductively coupled plasma spectroscopy method.

## 2 REFERENCES

The standards given below contain provisions which through reference in this text constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of these standards:

| *IS No.* | *Title* |
|---|---|
| IS 3025 | Methods of sampling and test (physical and chemical) for water and wastewater: |
| (Part 2) : 2019/ ISO 11885 : 2007 | Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP-OES) (*first revision*) |
| (Part 65) : 2022/ ISO 17294-2 : 2016 | Application of inductively coupled plasma mass spectrometry (ICP-MS) — Determination of selected elements including uranium isotopes (*first revision*) |
| IS 7022 (Part 1) : 1973 | Glossary of terms relating to water, sewage and industrial effluents: Part 1 |
| IS 7022 (Part 2) : 1979 | Glossary of terms relating to water, sewage and industrial effluents: Part 2 |
| IS 17614 | Water quality — Sampling: |
| (Part 1) : 2021/ ISO 5667-1 : 2020 | Guidance on the design of sampling programmes and sampling techniques |
| (Part 3) : 2021/ ISO 5667-3 : 2018 | Preservation and handling of water samples |

## 3 TERMINOLOGY

For the purpose of this standard, definitions given in IS 7022 (Part 1) and IS 7022 (Part 2) shall apply.

## 4 SAMPLING AND PRESERVATION

Sampling and sample preservation shall be done as prescribed in IS 17614 (Part 1) and IS 17614 (Part 3). Collect the sample in a polyethylene, polyvinyl chloride or chemically resistant glass container. Clean the sampling bottle with dilute nitric acid (6 N), prior to final rinsing with water and acidify the sample with nitric acid until the *pH* is approximately, but not less than 2. This acidification minimizes adsorption of manganese on the walls of the container and also assists in the dissolution of colloidal and particulate forms of manganese.

## 5 DIRECT AIR-ACETYLENE FLAME ATOMIC ABSORPTION SPECTROPHOTOMETRY METHOD

### 5.1 Principle

The manganese content of the sample is determined by atomic absorption spectrophotometry. For dissolved manganese, the filtered sample is directly aspirated to the atomizer. For total recoverable manganese, a pre-treatment with hydrochloric acid (HCl) is carried out prior to aspiration of the sample. This method is applicable in the range from 0.1 mg/l to 10 mg/l of manganese. However, the concentration range will vary with the sensitivity of the instrument used.

### 5.2 Interferences

**5.2.1** Add 1 g mercuric sulphate to prevent 0.1 g chloride in a 50 ml sample from interfering, as it forms slightly dissociated complexes. Samples that have been exposed to air may give low results due to precipitation of manganese dioxide ($MnO_2$). Add 1 drop of 30 percent hydrogen peroxide ($H_2O_2$) to the sample to re-dissolve precipitated manganese.

**5.2.2** Ferric ions form a violet complex which interferes with manganese determination. Add

EDTA and hydroxylammonium chloride/ammonia to reduce the interferences. The best method of overcoming this effect is to add a constant known amount of ammonium iron (II) sulphate to each calibration solution, blank test and test solution.

### 5.3 Apparatus

**5.3.1** *Atomic Absorption Spectrometer and Associated Equipment*

**5.3.2** Use burner head as recommended by the manufacturer

**5.3.3** *Standard Volumetric Glassware*

### 5.4 Reagents

**5.4.1** Unless otherwise specified, only AR grade chemicals should be used for all the tests.

**5.4.2** *Air* — clean, dried and free from oil, water and other foreign substances. The source may be a compressor or commercially bottled gas.

**5.4.3** *Acetylene* — standard commercial grade

**5.4.4** *Manganese* — free distilled water should be used for preparing standards and reagent solution.

**5.4.5** *Calcium Solution*

Dissolve 630 mg calcium carbonate ($CaCO_3$), in 50 ml of 1 + 5 HCl. Boil gently to obtain complete solution. Cool and dilute to 1 000 ml with water.

**5.4.6** *Hydrochloric Acid* (HCl) — 1 percent, 10 percent, 20 percent (all *v/v*), 1 + 5, 1 + 1, and concentrated.

**5.4.7** *Nitric Acid* ($HNO_3$) — 2 percent (all *v/v*), 1 + 5, 1 + 1, and concentrated.

**5.4.8** *Standard Manganese Solution*

Dissolve 0.100 g manganese metal in 10 ml concentrated HCl mixed with 1 ml concentrated $HNO_3$. Dilute to 1 000 ml with water (1.00 ml = 100 µg Mn).

### 5.5 Procedure

**5.5.1** *Sample Preparation*

Mix 25 ml of calcium solution (**5.4.5**) into 100 ml of sample solution before aspiration.

**5.5.2** *Instrument Operation*

**5.5.2.1** It is difficult to formulate instructions as applicable to every instrument, because of differences between makes and models of atomic absorptions spectrometers. See manufacturer’s operating manual.

**5.5.2.2** Install a hollow-cathode lamp for manganese in the instrument and set the wavelength at 279.5 nm. Set the slit width as suggested by manufacturer for manganese being measured. Turn on the instrument and apply current to hollow cathode lamp for 10 min to 20 min to stabilize the energy source. After adjusting the wavelength, install burner head (**5.3.2**). Turn on the air (**5.4.2**) and adjust the flow rate as suggested by the manufacturer to give maximum sensitivity for manganese being measured. Turn on acetylene (**5.4.3**) and adjust flow rate. Ignite the flame and let it stabilize for a few minutes.

**5.5.2.3** Aspirate a blank consisting of distilled water (**5.4.4**) containing 1.5 ml of concentrated nitric acid and 2 ml calcium solution. Set zero the instrument. Aspirate the manganese standard solution. Check the aspiration rate of nebulizer and adjust it to obtain maximum sensitivity. Obtain maximum response by adjusting burner position vertically and horizontally. Aspirate the blank and re-zero the instrument. Aspirate standard manganese solution near middle of linear range. Prepare a fresh same standard and with a new hollow cathode lamp, record the absorbance of this standard. Check consistency of instrument setup and aging of hollow-cathode lamp and standard referring to these data on subsequent determinations of manganese.

**5.5.2.4** The instrument is now ready to operate. At the end of analysis, first switch of acetylene than air.

**5.5.3** *Standardization*

Prepare at least three standard manganese solutions containing lower concentration 0.1 mg/l Mn to bracket the expected metal concentration of a sample. Prepare a reagent blank of 100 ml volume with distilled water (**5.4.4**) containing 1.5 ml of concentrated nitric acid (**5.4.7**) and 2 ml calcium solution (**5.4.5**). Aspirate the reagent blank and carry out zero adjustment. Aspirate blank and zero the instrument. Mix 2 ml of calcium solution (**5.4.5**) into the standard solutions before aspiration. Aspirate each standard solution in turn into the flame and record the absorbance readings. Most modern instruments are equipped with micro-processors and digital read-out which permit calibration in direct concentration terms.

**5.5.4** *Analysis of Sample*

Rinse nebulizer by aspirating distilled water (**5.4.4**) containing 1.5 ml concentrated nitric acid per litre and zero instrument. Aspirate a sample and determine its absorbance.

### 5.6 Calculation

Prepare a calibration curve on linear graph paper by plotting absorbance reading versus their reading. Alternatively, read the concentration directly from the instrument read out if the instrument is so equipped. If sample has been diluted, multiply by the appropriate dilution factor.

## 6 PER SULPHATE METHOD

### 6.1 Principle

In the presence of silver nitrate, permanganate is formed by persulfate oxidation of soluble manganous compounds. The resulting colour is stable for at least 24 h if excess persulfate is present and organic matter is absent.

### 6.2 Interference

**6.2.1** Interference of up to 0.1 gm chloride ($Cl^-$) in a 50 ml sample can be prevented by adding 1 gm mercuric sulphate ($HgSO_4$) to form slightly dissociated complexes. Trace amounts of bromide and iodide may interfere. The persulphate procedure is used for potable water with trace to small amounts of organic matter and gently heat after more persulfate has been added. For wastewaters containing organic matter, use preliminary digestion with nitric and sulphuric acids ($HNO_3$ and $H_2SO_4$). If large amounts of $Cl^-$ are present, boil by adding $HNO_3$. Traces of $Cl^-$ are eliminated by $HgSO_4$ in the special reagent. Coloured solutions from other inorganic ions are compensated during the final colorimetric step.

**6.2.2** Samples exposed to air may give low results due to precipitation of manganese dioxide ($MnO_2$). Hence, add 1 drop of 30 percent hydrogen peroxide ($H_2O_2$) after adding the special reagent to the sample for redissolving the precipitated manganese.

### 6.3 Minimum Detectable Concentration

The minimum detectable is concentration (98 percent transmittance) of 210 µg Mn/l when a 1 cm cell is used or 42 µg Mn/l when a 5 cm cell is used.

### 6.4 Apparatus

**6.4.1** *Colorimetric Equipment*

One of the following may be used:

**6.4.1.1** *Spectrophotometer* — for use at 525 nm, providing a light path of 1 cm or longer.

**6.4.1.2** *Filter photometer* — providing a light path of 1 cm or longer and equipped with a green filter having maximum transmittance near 525 nm.

**6.4.2** *Nessler Tube* — matched, 100 ml

### 6.5 Reagents

**6.5.1** *Special Reagent*

Dissolve 75 gm HgSO4 in 400 ml of concentrated $HNO_3$ and 200 ml of distilled water. Add 200 ml of 85 percent phosphoric acid ($H_3PO_4$), and 35 mg of silver nitrate ($AgNO_3$). Allow the solution to cool down and dilute the solution to 1 litre with water.

**6.5.2** *Ammonium Persulfate* [$(NH_4)_2S_2O_8$] — solid

**6.5.3** *Standard Manganese Solution*

**6.5.3.1** Prepare a 0.1 N potassium permanganate ($KMnO_4$) solution by dissolving 3.2 gm $KMnO_4$ in distilled water and making up to 1 litre. Allow the solution to undergo sunlight exposure for several weeks or heat it near the boiling point for several hours. Afterward, filter the solution through a fine fritted-glass filter crucible. To standardize the solution, use sodium oxalate as **6.5.3.2**.

**6.5.3.2** Weigh several 100 mg to 200 mg samples of $Na_2C_2O_4$, with a precision of 0.1 mg and transfer each sample to separate 400 ml beakers. To each beaker, add 100 ml distilled water and stir thoroughly to dissolve $Na_2C_2O_4$. Add 10 ml 1 + 1 $H_2SO_4$ to each beaker and rapidly heat the solutions to 90 °C to 95 °C. Titrate rapidly with the $KMnO_4$ solution to be standardized, while stirring, to a slight pink endpoint colour that persists for at least 1 min. Ensure that the temperature remains above 85 °C throughout the titration process. If necessary, warm the beaker contents during titration to maintain the desired temperature. Note that approximately 15 ml of permanganate solution will be consumed by 100 mg of $Na_2C_2O_4$. Perform a blank titration using distilled water and $H_2SO_4$ for comparison and calibration purposes.

$$\text{Normality of } KMnO_4,\ N = \frac{W}{(A - B) \times 0.06\,701}$$

where

$W$ = mass of $Na_2C_2O_4$, in mg;

$A$ = volume of titrant for sample, in ml; and

$B$ = volume of titrant for blank, in ml.

**6.5.3.3** Average the results of several titrations. Calculate volume of this solution necessary to prepare 1 litre of solution (1.00 ml = 50.0 µg of Mn), as follows:

$$\text{Volume of } KMnO_4,\ \text{ml} = \frac{4.55}{N}$$

where

N = normality of $KMnO_4$.

**6.5.3.4** To the solution, add 2 ml to 3 ml of concentrated $H_2SO_4$ and $NaHSO_3$ solution dropwise while stirring until the permanganate colour disappears. After that, boil the solution to eliminate any excess $SO_2$, then allow it to cool. Dilute the cooled solution to a final volume of 1 000 ml using distilled water. For measuring small amounts of manganese, further dilute this solution as needed.

**6.5.4** *Standard Manganese Solution* (*Alternate*)

Dissolve 1.000 gm of manganese metal (with a minimum purity of 99.8 percent) in 10 ml of redistilled $HNO_3$. Then, dilute the solution to a final volume of 1 000 ml using 1 percent (*v*/*v*) HCl (1 ml = 1.000 mg of Mn). For a more diluted solution, take 10 ml of the original solution and dilute it to 200 ml using distilled water (1 ml = 0.05 mg of Mn). Ensure to prepare the dilute solution daily for accurate measurements.

**6.5.5** *Hydrogen Peroxide* ($H_2O_2$) — 30 percent

**6.5.6** *Nitric Acid* ($HNO_3$) — concentrated

**6.5.7** *Sulfuric Acid* ($H_2SO_4$) — concentrated

**6.5.8** *Sodium Nitrite Solution*

Dissolve 5.0 gm $NaNO_2$ in 95 ml distilled water.

**6.5.9** *Sodium Oxalate* ($Na_2C_2O_4$) — primary standard

**6.5.10** *Sodium Bisulpite*

Dissolve 10 gm $NaHSO_3$ in 100 ml distilled water.

### 6.6 Procedure

**6.6.1** *Treatment of Sample*

If the sample is digested by using concentrated $HNO_3$ and concentrated $H_2SO_4$ for reducing organic matter or excessive chlorides, pipet a portion containing 0.05 mg to 2.0 mg Mn into a 250 ml conical flask. Makeup with distilled water up to 90 ml and proceed as given **6.6.2**.

**6.6.2** Take suitable sample portion, add 5 ml special reagent and 1 drop of $H_2O_2$. Concentrate to 90 ml by boiling or dilute to 90 ml. Add 1 gm $(NH_4)_2S_2O_8$, boil for 1 min. Do not heat on a water bath, cool under the tap. Boiling too long results in decomposition of excess persulphate and subsequent loss of permanganate colour; cooling too slowly has the same effect. Dilute to 100 ml with distilled water free from reducing substances and mix. Prepare standards containing 0 µg Mn, 5.00 µg Mn, ....1 500 µg Mn by treating various amounts of standard Mn solution in the same way.

**6.6.3** *Nessler Tube Comparison*

Use standards prepared as above and containing 5 µg Mn/100 ml to 100 µg Mn/100 ml final volume. Compare the samples and standards visually.

**6.6.4** *Photometric Determination*

**6.6.4.1** Prepare series of standards of 0 µg Mn/100 ml to 1 500 µg Mn/100 ml final volume. Make photometric measurements against a distilled water blank. The light path length appropriate for various amounts of manganese in 100 ml final volume as specified in Table 1.

**Table 1 Light Path Length Appropriate for Various Amounts of Manganese in 100 ml Final Volume**

(*Clause* 6.6.4.1)

| **Sl No.**<br>(1) | **Mn Range**, µg<br>(2) | **Light Path**, cm<br>(3) |
|---|---|---|
| i) | 5 to 200 | 15 |
| ii) | 20 to 400 | 5 |
| iii) | 50 to 1 000 | 2 |
| iv) | 100 to 1 500 | 1 |

**6.6.4.2** Prepare calibration curve of manganese concentration versus absorbance from the standards and determine the Mn concentration from the curve. If turbidity or interfering colour is present, make corrections as given in **6.6.4.3**. Correction for turbidity or interfering colour:

**6.6.4.3** Avoid filtration, as there is retention of some permanganate on the filter paper. If visual comparison is used, the effect of turbidity only can be estimated and no correction for colour interference.

**6.6.4.4** When photometric measurements are carried out made, use the following “bleaching” method, to remove colour interference. Soon after the photometer reading, add 0.05 ml $H_2O_2$ solution directly to the sample in the optical cell. Mix and as soon as the permanganate colour has faded completely and no bubbles remain, read again. Deduct absorbance of bleached solution from initial absorbance to obtain absorbance due to Mn.

### 6.7 Calculation

a) When entire original sample is taken for analysis:

Concentration of manganese, mg Mn/l = $\frac{C}{D} \times \frac{100}{E}$

where

$C$ = mass of manganese per 100 ml, µg Mn/100 ml;

$D$ = volume of sample, in ml; and

$E$ = volume of portion, in ml.

b) When a portion of the digested sample (100 ml final volume) is taken for analysis:

Concentration of manganese, mg Mn/l = $\frac{F}{G}$

where

$F$ = mass of manganese (in 100 mL final volume), in µg; and

$G$ = volume of sample, in ml.

## 7 INDUCED COUPLED PLASMA SPECTROSCOPY METHOD

Manganese can also be determined by induced coupled plasma optical emission spectroscopy with reference to procedure given in IS 3025 (Part 2). Likewise, induced coupled plasma mass spectroscopy with reference to procedure given in IS 3025 (Part 65) can also be used for the determination of manganese.

## ANNEX A

(*Foreword*)

## COMMITTEE COMPOSITION

Water Quality Sectional Committee, CHD 36

| *Organization* | *Representative(s)* |
|---|---|
| Chief Scientist, EPTRI, Hyderabad | Shri N. Raveendhar **(*Chairperson*)** |
| Andhra Pradesh Pollution Control Board, Vijaywada | Shrimati M. Sreeranjani<br>Shrimati A. Sri Samyuktha (*Alternate*) |
| Bhabha Atomic Research Centre, Mumbai | Dr S. K. Sahu<br>Shri I. V. Saradhi (*Alternate*) |
| Central Institute of Mining and Fuel Research, Dhanbad | Dr Abhay Kumar Singh |
| Central Pollution Control Board, New Delhi | Shri P. K. Mishra<br>Shri Vishal Gandhi (*Alternate*) |
| Confederation of Indian Industry, New Delhi | Dr Kapil K. Narula<br>Dr Sipika Chauhan (*Alternate*) |
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| FAD 14, Bureau of Indian Standards, New Delhi | Ms Nitasha Doger |
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Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the website-www.bis.gov.in or www.standardsbis.in.

This Indian Standard has been developed from Doc No.:CHD 36 (19331).

## Amendments Issued Since Publication

| Amend No. | Date of Issue | Text Affected |
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